

Use of betuline as a filler in paper and board**Field of the invention**

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The present invention relates to the use of betuline as a filler in paper and board, to a method for producing paper and board wherein betuline is used as a filler, and further, to paper and board comprising betuline as a filler.

10 **Prior art**

Mainly inorganic fillers such as kaolins, talc, silica, silicates, titanium dioxide, and calcium carbonates are used as fillers in paper and board, and moreover, to some extent fillers comprising organic polymer pigments which may also be hollow. As examples thereof, polymer pigments based on urea/formaldehyde resins may be mentioned.

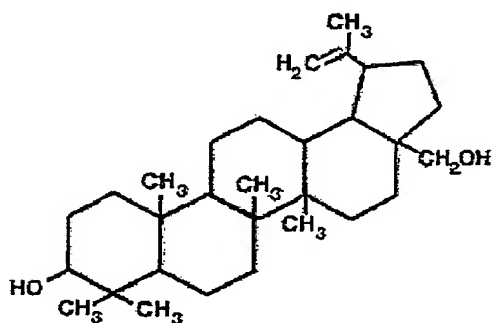
It is desired that the filler lowers the light scattering coefficient and tensile index of paper as little as possible. Among the inorganic fillers most commonly used, expensive titanium dioxide has an excellent light scattering coefficient.

Within the European Union is under consideration that in the future, it will be prohibited to dump compostable material to landfills. Then, the only economically realistic alternative for the disposal of compostable waste material will be incineration thereof. Inorganic pigments won't, however, burn, but produce ash residues as waste, whereas organic compounds may be disposed of by incineration without waste residues. Moreover, within the EU, goals set concerning the proportion of bioenergy in total energy production should be reached by 2010, and thus, combustible organic pigments are also desirable in this respect.

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Great amounts of birch bark waste is produced annually by chemical forest and sawmill industries, this waste being mainly disposed of by incineration. Betuline is found in birch bark, the content thereof in outer birch rind being about 20 – 40 %. Betuline may be isolated from birch, such as from the outer rind of the birch species *Betula verrucosa* common in Scandinavia by extracting with organic sol-
5 vents using methods known as such.

Betuline has a pentacyclic triterpene alcohol structure and it is also known by the trivial name betulinol and by the systematic name lup-20(29)ene-3,28-diol. The
10 structure of betuline is shown by the following formula I:



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Betuline is a white crystalline powder with a low density having a strongly hydro-
15 phobic nature, and it is hardly soluble in water.

Use of betuline in paper industry has been studied to some extent, particularly its effect on the yellowing of paper, brightness reversion of pulp, and its odour. In *FI 10402*, use of betuline as a coating pigment of paper and board either alone or
20 together with other pigments is discussed. A coating paste was made of betuline by slurring it together with excipients in water in a similar manner as with talc and by stabilizing the slurry with a stabilizer. Coating pastes were prepared from the slurry thus obtained using excipients known in the art, either with or without other coating pigments such as kaolin, calcium carbonate or talc. With this coating
25 paste containing betuline, coated base papers having an improved light scattering

could be made because of the fact that in the structure of betuline, there are, among others, hydroxyl groups necessary for the carriers of optical brighteners.

On the basis of the above discussion, it may be seen that there is a need for papers
5 and boards made from combustible raw materials, the properties thereof, however, corresponding to those of papers and boards prepared with conventional fillers, as well as for a method for producing said papers and boards.

Disclosure of the invention

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The invention is directed to the use of betuline as a filler in paper and board.

The invention also relates to a method for producing paper and board, comprising the use of betuline as a filler.

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Moreover, the invention relates to papers and boards containing betuline as a filler.

Characteristic features of the use of betuline in the production of paper and board,
20 the method for producing paper and board, and papers and boards containing betuline as a filler according to the invention are presented in the claims.

Brief description of the invention

25 It was surprisingly found that problems and drawbacks of known solutions of prior art may be solved or at least essentially avoided by using betuline as a filler in paper and board. In the production of paper or board, betuline slurried in water is added to the pulp, preferably after the addition of retention aids, that is, as an aqueous slurry preferably containing betuline no more than 60 % by weight, particularly preferably from 5 to 35 % by weight. Thus, paper and board that may be
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incinerated and having properties corresponding to those of papers and boards yet produced with conventional fillers may be obtained.

Detailed description of the invention

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According to the invention, betuline may be used as a filler in paper and board, particularly as a filler in uncoated paper and board, preferably alone, but also together with known inorganic and/or organic fillers. An aqueous slurry is prepared from betuline and then, this slurry is added to the pulp after the addition of retention aids during the production of paper or board. Preferably, no more than 60 %
10 by weight, particularly preferably from 5 to 35 % by weight of betuline is dispersed in water with one or more excipients. One or more conventional agents used for dispersion of hydrophobic compounds may be used as excipients. Said adjuvants are preferably selected from a group consisting of dispersing agents,
15 surface active agents and stabilizers that stabilize the dispersion and prevent the formation of agglomerates.

Dispersing agent may be used in an amount ranging from 0 to 1 % by weight, preferably from 0.01 to 0.4 % by weight, the amount of the surface active agent being
20 0 to 6 % by weight, preferably from 2.5 to 5 % by weight, and that of the stabilizer being from 0 to 1 % by weight, preferably from 0.1 to 0.3 % by weight. Useful dispersing agents include dispersing agents suitable for hydrophilic fillers such as salts of the acrylic acid polymers, examples of suitable surface active agents include polyethylene glycols, and suitable stabilizers include polyvinyl alcohols.
25 Percentages by weight are calculated relative to the slurry.

If necessary, the pH of the slurry is adjusted with a base to a value of at least 8.5, preferably between 8.5 and 10. Examples of suitable bases include inorganic bases such as NaOH, KOH and the like.

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Betuline is preferably used as a fine powder having a mean particle size of no more than 30 μm , the particle size being preferably between 0.3 and 10 μm , particularly preferably between 0.5 and 2.5 μm . A SEM image of milled betuline particles is presented in the appended figure 1.

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Betuline may be betuline prepared from birch, that is betuline isolated for instance by extraction from birch bark, from bark waste produced by pulp plants or industrial sawmills, or it may be synthetically produced betuline. Betuline having a purity of at least 85 % by weight, preferably at least 95 % by weight, is preferably used.

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The method of the invention for producing paper or board comprises steps wherein an aqueous slurry is prepared containing preferably no more than 60 % by weight, particularly from 5 to 35 % by weight of fine betuline that is adjusted to a desirable particle size for instance by milling, if necessary, and then said slurry is added to the pulp used for paper or board production, particularly to fine paper pulp, preferably after the addition of retention aids. Then water is removed from the paper web followed by drying and calendering the paper or board if necessary, that is, the production of paper or board is continued in a conventional manner known as such to those skilled in the art.

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Pulp used in the production of paper or board comprises chemical pulp or mechanical pulp or a mixture thereof, preferably pulp for fine papers including softwood and hardwood pulps. Different pulps may be freely mixed depending on the product to be produced.

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The paper or board of the invention comprises betuline as the filler, the amount of betuline in the paper or board ranging between 0.1 and 60 %, preferably between 5 and 35 % by weight.

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Betuline may be used as a filler in fine papers, in papers containing chemical and mechanical pulps, in uncoated and coated fine papers, in coated papers containing mechanical pulp, and moreover in the surface layers of boards, for instance in folding boxboard and linerboard. Preferably betuline is used in fine papers containing at present high amounts of inorganic fillers in conventional manner.

The present invention has considerable advantages compared to prior art. Retention of betuline is clearly higher than that of traditional inorganic fillers. Due to this high retention of betuline, the quality of recycled water is improved and purer recycled water is obtained and thus, lower amounts of fixing agents and retention aids are necessary. Formation is improved due to the reduction of retention aids. In addition, the examples show that in paper types containing betuline as a filler, the combination of tensile index and light scattering is clearly improved, and the tensile index may be increased in comparison to precipitated calcium carbonate by about 40 % without lowering the light scattering coefficient. This is illustrated by figure 2. Interlaminar strength of paper and board represented by Scott bond value is also somewhat increased.

Compared to the known precipitated calcium carbonate (PCC) filler providing high bulk, betuline surprisingly renders high bulk but low porosity. Due to this low porosity, an impervious paper for coating having a superior surface is obtained, and accordingly during the coating step of the paper, clearly less coating paste is needed for high coating power. Moreover, betuline prevents the pulp from yellowing as shown by the results of Example 3.

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The density of betuline being low, it is a light-weight substance, whereas traditional inorganic pigments are heavy causing clearly higher logistic costs than light-weight substances. With this light betuline, lighter printing and news quality papers may be produced, thus reducing mailing and other logistic costs and costs due to waste paper, and accordingly, less waste detrimental to the environment is produced.

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Unlike betuline, inorganic pigments do not belong to renewable resources. Betuline is an organic compound having a high heat value when burned, and further, it does not produce ash. In addition, betuline has antiviral, antifungal and antimicrobial activities, and thus the amounts of antimicrobial agents used in the production of paper and board may be reduced, which clearly lowers the environmental burden caused by said agents.

The invention is now illustrated with the following examples without limiting the scope of the invention thereto.

Examples

Example 1

Dispersion of betuline to give a slurry

Betuline was milled to give a fine powder having a mean particle size of about 1 μm . Figure 1 shows the SEM image of the milled betuline. Then, 0.15 parts by weight of a dispersing agent (Fennodispo A41), 3.5 parts by weight of a surface active agent (Lutesol T07), and 0.02 parts by weight of a 10 % NaOH solution were added to water and mixed, followed by the addition of 100 parts by weight of the milled betuline and 0.15 parts by weight of a stabilizer (Celvol 103) to the mixture thus obtained. Then, the mixture was slurried for 40 minutes using a Diaf dispersing apparatus.

Example 2

Preparation and testing of sheets containing betuline

For producing the sheets, 70 % by weight of SR 21 birch pulp and 30 % by weight of SR 27 softwood pulp were used. 0.3 mg/g of the retention aid Percol 47, relative to the absolute dry weight of the pulp, and from 5 to 30 % by weight of the

betuline dispersion prepared in Example 1 were added to the pulp mixture. From the pulp thus obtained, normal laboratory sheets having a surface weight of 60 g/m² were made, compressed in conventional manner, dried and tested.

- 5 The sheets were extracted with acetone, followed by the determination of the amount of betuline by gas chromatography using an internal standard. Retention of betuline was rather high, the filler contents being 10 % and 20 %. Control sheet 1 contained no filler, precipitated calcium carbonate (PCC) in scalenohedron form was used in other control sheets. The retention of PCC was lower, and at best,
- 10 filler contents were 8 % and 3 %. When comparing sheets containing 10 % of betuline and 8 % of PCC, it was found that betuline gave better strenght results, particularly the tensile index being clearly higher. Moreover, the porosity was better with betuline compared to control agents. The results are shown in Tables 1 and 2 below, and in the appended Figure 2, the tensile index is presented as the
- 15 function of the light scattering coefficient.

Table 1

Sample	Grammage, g/m ²	Thickness, μ m	Bulk, cm ³ /kg	Tensile index, kNm/kg	Interlaminar strenght / Scott bond, J/m ²	Porosity, ml/min
Control 1	59.5	76	1.27	49.0	450	470
Betuline 10 %	64.8	107	1.64	71.5	300	950
Betuline 20 %	72.5	135	1.86	28.8	130	1350
PCC 3 %	57.2	77	1.34	57.5	415	790
PCC 8 %	59.1	82	1.39	52.0	285	1030

Table 2

Sample	Grammage, g/m ²	Opacity, %	Light scattering coefficient, m ² /kg
Control 1	59.5	63.8	24.8
Betuline 10 %	64.8	77.1	37.6
Betuline 20 %	72.3	86.2	53.5
PCC 3 %	57.2	68.3	31.0
PCC 8 %	59.1	73.3	37.9

Example 3**5 Preparation and testing of sheets containing betuline**

Laboratory sheets having a surface weight of 60 g/m² were prepared from chemical pulps containing respectively 10 and 20 % by weight of betuline, and for comparison purposes, 3 and 8 % by weight of PCC filler (Alcabar LO), the composition of the pulp being 70 % by weight of birch and 30 % by weight of tall.

Figures 3 and 4 show the results of the ISO brightness test and absorption coefficient test. The sheets containing betuline turned yellow clearly slower than sheets containing PCC filler, and further, the sheets containing betuline were whitened more readily by light than other sheets. With respect to optical properties, the sheets containing betuline corresponded to sheets containing the PCC filler.